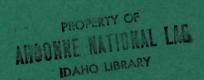
THE VISCOSITY OF MOLTEN ALUMINA

by

R. A. Blomquist, J. K. Fink, and L. Leibowitz



BASE TECHNOLOGY



ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

Prepared for the U. S. DEPARTMENT OF ENERGY
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Printed in the United States of America
Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161
Price: Printed Copy \$4.00; Microfiche \$3.00

ANL-78-28

ARGONNE NATIONAL LABORATORY 9700 South Cass Avenue Argonne, Illinois 60439

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Chemical Engineering Division

March 1978

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THE VISCOSITY OF MOLTEN ALUMINA

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ABSTRACT

In the analysis of LMFBR core-containment and heat-removal problems associated with hypothetical core-disruptive accidents, viscosity data on molten ceramics are needed to help analyze the convective heat transfer and flow patterns within liquid pools. An oscillating cup viscometer has been used to measure the viscosity of molten alumina in the temperature range from 2400 to 2750 K. The data are represented by the equation:

$$\log \eta = \frac{11448}{T} - 8.2734$$

where the viscosity, η , is given in pascal seconds and the temperature, T, is in kelvins.

I. INTRODUCTION

Viscosity data on molten ceramics are needed in the analysis of the core-containment and heat-removal problems associated with hypothetical core-disruptive accidents in liquid-metal fast breeder reactors. Specifically, viscosity data are needed to analyze the behavior of sacrificial beds in modeling studies of post-accident heat removal, because the viscosities of molten ceramics would affect convective heat transfer and flow patterns within liquid pools. Initial measurements on the viscosity of molten alumina in the temperature range 2400 to 2750 K are reported here.

The methods available for the measurement of the viscosity of sacrificial barrier materials are limited by the high temperatures and relatively low viscosities that are anticipated in these experiments. The oscillating cup viscometer is an excellent means for making measurements under these conditions because it allows measurements to be made on materials with a variety of properties, such as high vapor pressure, high melting point, and significant chemical reactivity.

The oscillating cup viscometer consists of a torsionally oscillating closed cup, containing the material of interest, suspended from a wire. From the measured logarithmic decrement of decay of the torsional oscillations of the cup, the viscosity can be calculated.

II. THEORETICAL

A solution of the equations of motion for a torsionally oscillating, damped harmonic oscillator has been presented by Finucane and Olander. In the dimensionless parameters introduced by Wittenberg, Ofte, and Curtiss, the final equation is:

$$4\pi\delta + Q[\delta Im(F) - 2\pi Re(F)] = 0$$
 (1)

where δ is the logarithmic decrement of the amplitude attenuation. The term Q is represented by

$$Q = \frac{1}{2} \cdot \frac{ma^2}{I} \tag{2}$$

where

m = mass of liquid in the crucible

a = radius of the crucible

I = moment of inertia of the empty system

The terms $\operatorname{Im}(F)$ and $\operatorname{Re}(F)$ denote the imaginary and real parts of the complex function

$$F = \left(\frac{4\lambda v\tau}{a^2}\right) \left[S_1(\lambda) + 2BS_2(\lambda)\right]$$
 (3)

where

$$\lambda^2 = \frac{a^2}{v\tau}(2\pi i - \delta) \tag{4}$$

v = kinematic viscosity

 τ = period of oscillation

and

$$B = a/H \tag{5}$$

where

H = height of the liquid in the crucible

The terms $S_1(\lambda)$ and $S_2(\lambda)$ in the function F have been shown by $Roscoe^3$ to be approximated by the series:

$$S_1(\lambda) = 1 - \frac{3}{2\lambda} + \frac{3}{8\lambda^2} + \frac{3}{8\lambda^3} + \frac{63}{128\lambda^4} + \dots$$
 (6)

$$S_2(\lambda) = \frac{1}{8} - \frac{2}{\pi^{\lambda}} + \frac{9}{8\lambda^2} - \frac{1}{\pi^{\lambda^3}} - \frac{45}{128\lambda^4} + \dots$$
 (7)

With all of the parameters except ν specified, Eq. 1 can be solved by trial and error insertion of values for ν . It would, however, be much more convenient if we could find an exact solution for ν . In Eq. 4,

1et

$$\omega^2 = \frac{a^2}{\tau} (2\pi i - \delta) \tag{8}$$

then

$$\lambda = \frac{1}{v^{1/2}} \quad \omega \tag{9}$$

Using this definition of λ Eq. 3 becomes

$$F = \frac{4\omega v^{1/2} \tau}{a^2} \left[S_1 \left(\omega v^{-1/2} \right) + 2BS_2 \left(\omega v^{-1/2} \right) \right]$$
 (10)

and Eqs. 6 and 7 become

$$S_1(\omega v^{-1/2}) = 1 - \frac{3}{2\omega} v^{1/2} + \frac{3}{8\omega^2} v + \frac{3}{8\omega^3} v^{3/2} + \frac{63}{128\omega^4} v^2 + \dots$$
 (11)

$$S_2(\omega v^{-1/2}) = \frac{1}{8} - \frac{2}{\pi \omega} v^{1/2} + \frac{9}{8\omega^2} v - \frac{1}{\pi \omega^3} v^{3/2} - \frac{45}{128v^4} v^2 + \dots$$
 (12)

Let

$$z = v^{1/2},$$

then Eqs. 10, 11, and 12 become

$$F = \frac{4\omega\tau Z}{a^2} \left\{ \left[1 - \frac{3Z}{2\omega} + \frac{3Z^2}{8\omega^2} + \frac{3Z^3}{8\omega^3} + \frac{63Z^4}{128\omega^4} \right] + 2B \left[\frac{1}{8} - \frac{2Z}{\pi\omega} + \frac{9Z^2}{8\omega^2} - \frac{Z^3}{\pi\omega^3} - \frac{45Z^4}{128\omega^4} \right] \right\}$$
 (13)

Combining powers of Z

$$F = \frac{4\omega\tau Z}{a^2} \left\{ [1 + 2B(1/8)] - \left[\frac{3}{2\omega} + 2B\left(\frac{2}{\pi\omega}\right) \right] Z + \left[\frac{3}{8\omega^2} + 2B\left(\frac{9}{8\omega^2}\right) \right] Z^2 + \left[\frac{3}{8\omega^3} - 2B\left(\frac{1}{\pi\omega^3}\right) \right] Z^3 + \left[\frac{63}{128\omega^4} - 2B\left(\frac{45}{128\omega^4}\right) \right] Z^4 \right\}$$
(14)

Let

$$F_1 = \frac{4\omega\tau}{a^2} \left(1 + \frac{2B}{8} \right) \tag{15}$$

$$F_2 = \frac{4\omega\tau}{a^2} \left[\frac{3}{2\omega} + 2B \left(\frac{2}{\pi\omega} \right) \right]$$
 (16)

$$F_3 = \frac{4\omega\tau}{a^2} \left[\frac{3}{8\omega^2} + 2B \left(\frac{9}{8\omega^2} \right) \right] \tag{17}$$

$$F_{4} = \frac{4\omega\tau}{a^2} \left[\frac{3}{8\omega^3} - 2B \left(\frac{1}{\pi\omega^3} \right) \right] \tag{18}$$

$$F_5 = \frac{4\omega\tau}{a^2} \left[\frac{63}{128\omega^4} - 2B \left(\frac{45}{128\omega^4} \right) \right]$$
 (19)

then

$$F = F_1 Z + F_2 Z^2 + F_3 Z^3 + F_4 Z^4 + F_5 Z^5$$
 (20)

which is a power series in Z:

$$F = \sum_{i=1}^{5} F_{i}Z^{i}$$
 (21)

Eq. 1 now becomes

$$4\pi\delta + Q \sum_{i=1}^{5} [\delta Im(F_i) - 2\pi Re(F_i)] Z^i = 0$$
 (22)

which is a 5th order polynomial in Z. The roots of the polynomial were found using standard numerical techniques. The kinematic viscosity $\nu = Z^2$ could then be calculated. Of the five roots which satisfied Eq. 22, three were rejected because they would not have any physical significance. Two of these roots were complex and one was negative. The negative root was rejected because, in boundary-layer theory, the boundary-layer thickness is proportional to Z, the square root of the viscosity. A negative square root would not have any physical significance.

When the two remaining roots were plotted as a function of temperature, one root increased with temperature and the other decreased. In Newtonian liquids, the viscosity decreases logarithmically as the temperature is increased, therefore, the root that increased with temperature was discarded and the kinematic viscosity was calculated using the remaining root. The absolute viscosity was then calculated from the kinematic viscosity and the density, ρ , as follows:

$$\mu = \rho \nu \tag{23}$$

III. EXPERIMENTAL

The oscillating cup viscometer as shown in Fig. 1 consists of a closed cup suspended from a tungsten wire. The cup (Fig. 2) is a tungsten crucible 25.48 mm in diameter and 50.8 mm high. In the center of the bottom is a blackbody cavity 0.508 mm in diameter and 3.05 mm deep. The filling neck is approximately 76.2 mm long by 7.49 mm ID. All wall thicknesses are 1.02 mm. The crucible is a one-piece unit commercially fabricated by chemical vapor deposition. After the crucible is filled, a solid tungsten plug is slip-fitted into the filling neck and electron-beam-welded in place.

The sealed crucible is attached to a cylindrical block (an "inertia block") by means of a tantalum connecting rod. The inertia block is designed to give the oscillating system a reasonable rate of attenuation and provide support for the additional inertia blocks used to measure the moment of inertia. Above the inertia block is attached a rod containing a reflecting mirror, which is attached to the suspension wire by means of a small chuck. The tungsten suspension wire is 0.178 mm in diameter by 0.31 m long. The upper end of the wire is attached by a chuck to a rotational mechanical feedthrough. Rotation of the feedthrough is used to start oscillation of the suspended system.

The combined parameters of wire material and diameter, and the inertia block mass and diameter significantly affect the rate of attenuation of the suspended system. If the rate is too high, flow within the crucible is turbulent and erroneous measurements may result; if rate is too low, an unreasonably long time may be required to make a measurement.

The entire assembly was mounted in a high vacuum, ion-pumped, tungsten mesh furnace (Varian VF-100). The hot zone of the furnace consists of a split tungsten-mesh heating element with power provided by a stepdown transformer. A front-opening door allows complete access to the work zone of the furnace. Temperature control was achieved by monitoring the power input through a fast response thermal-watt converter and controlling the power input, either manually or automatically.

A vacuum of 3 x 10^{-7} Pa was achieved through a combination of mechanical, titanium-sublimation, and ion pumps. Before measurements were performed, the mechanical pump was turned off. The ion pump and sublimation pump then provided a vibration-free pumping system for the furnace.

The logarithmic decrement of the oscillating cup is measured by shining a narrow beam of light from a small helium-neon laser through a window onto the reflecting mirror, as shown in Fig. 3. The reflected light beam strikes

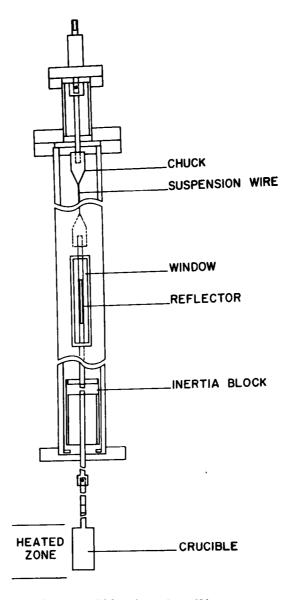


Fig. 1. Oscillating Cup Viscometer ANL Neg. No. 308-77-210

a photodetector each time the suspension system passes a precisely known point slightly offset from center. The time interval for each successive swing in the same direction is measured with a Monsanto 8510 counter/timer and is recorded on an attached printer. The logarithmic decrement, δ , is calculated from the relationship

$$\delta = \frac{1}{N} \ln \frac{\cos \left[\frac{(\Delta t)_{N} \pi}{\tau} \right]}{\cos \left[\frac{(\Delta t)_{0} \pi}{\tau} \right]}$$

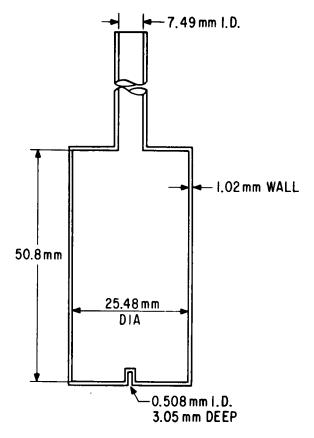


Fig. 2. Tungsten Viscometer Cup ANL Neg. No. 308-77-636

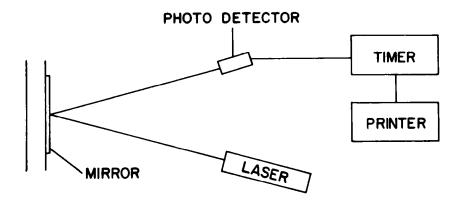


Fig. 3. Schematic of Electronic Equipment for Viscosity Measurements ANL Neg. No. 308-77-211

where

 $(\Delta t)_{N}$ = the time interval of the Nth swing

 $(\Delta t)_0$ = the time interval of the initial swing

N = number of swings measured

 τ = period of the oscillations

When measurements are made, the logarithmic decrement is calculated for each set of 10 successive swings. Usually, 10-20 logarithmic decrement measurements are made at a given temperature and are then averaged to obtain a final value.

The temperature of the crucible was measured using a Leeds and Northrup Model 8634-C disappearing-filament optical pyrometer. The optical pyrometer was sighted through a prism and window located at the bottom of the furnace onto the blackbody cavity in the sample crucible. A magnetic shutter protected the window from exposure to the furnace environment except while temperature measurements were being made. The optical pyrometer, window, and prism were calibrated with strip lamps which had been calibrated by the National Physical Laboratory (Teddington, England).

The material used in these measurements was alumina powder of 99.99% purity obtained from Alfa Division, Ventron Corporation. The crucible was filled incrementally with a known amount of alumina by successive loading and melting steps. The tungsten plug was then electron-beam-welded in place under vacuum to complete preparations for the measurements.

Spectrochemical analysis of the alumina before and after these measurements showed no significant changes in composition. The tungsten concentration was <0.01% before and ~0.01 % after the measurements. These results indicate that essentially no interaction occurred between the alumina and the crucible.

IV. RESULTS AND DISCUSSION

To calculate viscosity from the logarithmic decrement, it is first necessary to measure the inherent logarithmic decrement and the moment of inertia. The inherent logarithmic decrement is due primarily to the restoring force caused by the internal friction within the twisting suspension wire. Before measurements were made on molten alumina, several measurements of the logarithmic decrement were made just below the melting point of alumina. The average of these measurements was 0.001577 and this was subtracted from the logarithmic decrements measured for molten alumina.

Using the fact that the period varies inversely with the square root of the moment of inertia, the moment of inertia for the whole system was determined by measuring the period of the system and then adding several blocks with precisely known moments of inertia. From these measurements, the moment of inertia was found to be $2.4001 \times 10^{-4} \text{ kg} \cdot \text{m}^2$.

In order to test operation of the system, viscosity measurements were made on water at room temperature. Results agreed within an average of 2% with literature values. This agreement was considered satisfactory for our purposes.

The viscosity of molten alumina was measured in the temperature range from 2394 to 2742 K. The results of the viscosity measurements are given in Table 1. At each temperature the corrected radius of the crucible was calculated using the thermal expansion data of Touloukian. The density of alumina used to calculate viscosities is the average of the densities measured by Bates and Elyutin, whose values agreed within 1.5%. Over the temperature range of our experiments, the viscosity, n, may be represented by the equation:

$$\ln \eta = \frac{11448}{T} - 8.2734$$

where η is in pascal seconds* and the temperature T is in kelvins. The estimate of the standard error of this fit is about 4%. Our viscosity data are presented graphically in Fig. 4. Also presented in Fig. 4 for comparison are the viscosity data of Bates⁶ and of Elyutin.⁷ Our data agree well with those of Elyutin but differ significantly from those of Bates. The cause of this discrepancy is not known at present. The activation energy for viscous flow calculated from our data is 95 kJ/mol. This value is in fair agreement with the value of 126 kJ/mol reported by Elyutin.⁷

Table 1. Measurements of Viscosity of Alumina

Mass of Alumina: 57.365 gMoment of Inertia: $2.4001 \times 10^{-4} \text{ kg} \cdot \text{m}^2$

Crucible Radius at 25°C: 12.74 mm

Temp., K	Density, 10 ³ kg/m ³	Period, s	Logarithmic Decrement	Viscosity Pa·s		
2458	2.85	15.297	0.0144	0.0264		
2509	2.79	15.303	0.0153	0.0243		
2412	2.90	15.305	0.0131	0.0288		
2558	2.73	15.282	0.0166	0.0222		
2661	2.62	15.303	0.0181	0.0186		
2742	2.52	15.303	0.0190	0.0164		
2618	2.67	15.302	0.0167	0.0216		
2398	2.92	15.287	0.0121	0.0307		
2453	2.85	15.294	0.0149	0.0255		
2420	2.89	15.293	0.0124	0.0300		
2394	2.92	15.293	0.0117	0.0313		

 $^{^*1 \}text{ Pa·s} = 10^3 \text{ cp.}$

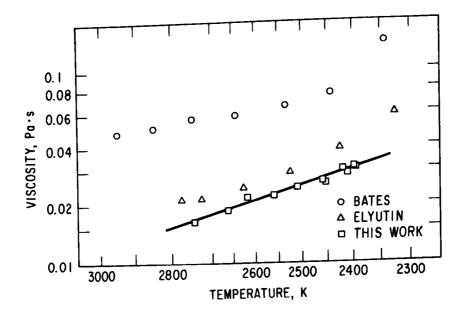


Fig. 4. Viscosity of Molten Alumina ANL Neg. No. 308-77-421

Several investigators⁸⁻¹² have suggested empirical relationships which can be used to estimate the viscosity of molten materials near their melting points. These methods have been applied to molten alumina, and the results are summarized in Table 2. The wide range of estimates given in the table indicates the difficulty that can be encountered in accurately estimating the viscosity of a material such as molten alumina. Most empirical methods of estimation are based on materials such as metals, which have less complex molecular structures than alumina, and it is probably for this reason that these estimation methods fail when applied to molten alumina.

Table 2. Viscosity of Molten Alumina Near the Melting Point: Experimental and Calculated Values

	Viscosity, Pa·s
Bird, Stewart, and Lightfoot (Ref. 8)	0.0056ª
Glasstone et al. (Ref. 9)	0.343 ^a
Partington (Ref. 10)	0.00237 ^a
Kumar et al . (Ref. 11)	0.003 ^b
Sokolov (Ref. 12)	9.43 ^b
Experimental, this work	0.035

^aCalculated by us using the method described in the reference given.

b_Estimated value from the reference given.

V. SUMMARY

The viscosity of molten alumina has been measured using an oscillating cup viscometer. The viscosity, η , is represented by the equation

$$\ln \eta = \frac{11448}{T} - 8.2734$$

for the the temperature range from 2394 to 2742 K. The activation energy for viscous flow is 95 kJ/mol.

The experimental measurements on molten alumina have demonstrated the usefulness of the oscillating cup method for measuring the viscosity of materials at high temperatures.

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